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IS 15556 (2005): Volumetric and spectrophotometric estimation of iron (Amalgamating IS 4264:1967, IS 4542:1968) [CHD 1: Inorganic Chemicals]

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भारतीय मानक

लोहे का आयतनी और स्पेक्ट्रोमीटरी आकलन

Indian Standard

VOLUMETRIC AND SPECTROPHOTOMETRIC ESTIMATION OF IRON

ICS 71.040.50, 77.080.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2005 **Price Group 3**

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This standard has been formulated by amalgamating IS 4284 'Method for volumetric determination of iron' and IS 4542 'Colorimetric methods for determination of iron'. IS 4284 was published in 1967 whereas IS 4542 was published in 1968. Volumetric methods of analysis of iron had been incorporated in IS 4284 and spectrophotometric methods in IS 4542. The objective of the two standards was to achieve uniformity of the method of volumetric and spectrophotometric determination of iron when prescribed in Indian Standards for analysis of chemical products.

Volumetric methods for determination of iron involve reduction of ferric to ferrous and titrating with standard potassium permanganate or standard potassium dichromate.

A number of organic reagents have been recommended for spectrophotometric analysis of iron. The basis of determination in each case is the comparison of colour developed by the reagents with iron with a set of known standards by means of a spectrophotometer. IS 4542 had therefore been proposed so as to bring about a uniformity in procedure for the application of organic reagents for the determination of iron.

The Committee agreed that as far as possible methods of test estimation of elements/compounds in different product standard should be harmonized. Keeping this in view, the Committee decided to amalgamate IS 4284 and IS 4542 for determination of iron incorporating both instrumental as well as non-instrumental methods after appropriate revision.

Volumetric methods described in IS 4284 shall be retained for estimation of iron content present in amount greater than 2 percent, in samples.

The spectroscopic methods described in IS 4542 were evaluated and the methods based on thioglycolic acid and 1-10, phenanthroline have been adopted as standard techniques. These techniques are widely used in the various standards.

The thiocyanate method suffers from several drawbacks; the colour is somewhat unstable, the stoichiometry is uncertain, and Beer's law is not strictly followed, hence not included in the standard.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final values, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

VOLUMETRIC AND SPECTROPHOTOMETRIC ESTIMATION OF IRON

1 SCOPE

This standard prescribes the method for volumetric determination of iron by using (a) potassium permanganate, and (b) potassium dichromate and spectrophotometric method for determination of iron by using (1) thioglycolic acid, and (2) *o*-phenanthroline.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2316:1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*second revision*)

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 VOLUMETRIC METHODS

4.1 Method A — Potassium Permanganate Method

4.1.1 *Reagents*

4.1.1.1 *Standard potassium permanganate solution* — 0.1 N (*see* IS 2316).

4.1.1.2 *Zimmermann-Reinhardt solution* — Add 140 ml of concentrated sulphuric acid and 140 ml of phosphoric acid (85 percent *w/w*) to 720 ml of water. Dissolve 200 g of hydrated manganese sulphate ($MnSO_4 . 4H_2O$) in this solution. While the solution is still hot, add potassium permanganate solution (0.1 N) drop by drop with stirring till the faint persistent pink colour is obtained.

4.1.1.3 *Dilute hydrochloric acid* — 1 : 1 (*v/v*).

4.1.1.4 *Stannous chloride solution* — Dissolve by heating 60 g of pure anhydrous stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is clear. Cool, add a few pieces of granulated tin and preserve the solution in an airtight amber-coloured bottle to prevent oxidation.

4.1.1.5 *Mercuric chloride solution* — Prepare a saturated solution of mercuric chloride in water.

4.1.2 *Procedure*

4.1.2.1 Pipette 50 ml of the clear solution of the material to be analyzed as prepared in the relevant specification into a 500 ml conical flask. This solution shall contain about 50 mg of iron. Add 10 ml of dilute hydrochloric acid and bring to boil. Evaporate to 40 ml and add 5 ml of concentrated hydrochloric acid. Reduce the iron by adding stannous chloride solution drop-wise until the solution becomes colourless. Then add 2 to 3 drops in excess. Cool the solution rapidly to room temperature under running tap water. Add 10 ml of mercuric chloride solution all at a time and stir thoroughly. A small silky white precipitate of mercurous chloride should form slowly, indicating that a slight excess of stannous chloride is present. Allow to stand for 2 to 5 min until no more precipitate appears to form.

4.1.2.2 To the solution, add 20 ml of Zimmermann-Reinhardt solution and dilute to 200 ml. Wash down the sides of the flask with distilled water and titrate with standard potassium permanganate solution to a faint pink end point which persists for 30 s.

4.1.3 *Calculation*

$$\text{Iron (as Fe), percent by weight} = \frac{A \times N \times 5.584}{W}$$

where

A = volume, in ml, of standard potassium permanganate solution required to titrate the test solution,

N = normality of standard potassium permanganate solution, and

W = weight, in g, of original material contained in the test solution.

4.2 Method B — Potassium Dichromate Method

4.2.1 *Reagents*

4.2.1.1 *Dilute hydrochloric acid* — *See* **4.1.1.3**.

4.2.1.2 *Stannous chloride solution* — *See* **4.1.1.4**.

4.2.1.3 *Mercuric chloride solution* — *See* **4.1.1.5**.

4.2.1.4 *Dilute potassium permanganate solution*, approximately 0.1 N.

4.2.1.5 *Sulphuric acid — Phosphoric acid mixture* — Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this 150 ml of phosphoric acid (85 percent *w/w*). Destroy any oxidizable impurities by adding potassium permanganate solution (0.1 N) drop by drop until the pink colour of permanganate persists, and cool the solution.

CAUTION — Concentrated sulphuric acid is highly corrosive, use appropriate safety implements.

4.2.1.6 *Sodium diphenylamine sulphonate indicator solution* — Dissolve 0.2 to 0.3 g of sodium diphenylamine sulphonate in 100 ml of hot water. Cool and store in amber coloured bottle.

4.2.1.7 *Standard potassium dichromate solution* — 0.1 N (*see* IS 2316).

4.2.2 *Procedure*

4.2.2.1 Proceed exactly as given in **4.1.2.1**.

4.2.2.2 Add 15 ml of sulphuric acid-phosphoric acid mixture and dilute to 100 ml. Add 5 drops of sodium diphenylamine sulphonate indicator solution and titrate with standard dichromate solution while stirring continuously until the green colour begins to darken. Continue titration until a drop of dichromate solution produces a permanent violet-blue colour. Determine indicator blank using same amount of reagents.

4.2.3 *Calculation*

$$\text{Iron (as Fe), percent by weight} = \frac{(A-B) \times N \times 5.584}{W}$$

where

A = volume, in ml, of standard potassium dichromate solution required to titrate the test solution;

B = volume, in ml, of standard potassium dichromate solution required for the blank;

N = normality of the standard potassium dichromate solution; and

W = weight, in g, of material contained in the test solution.

5 SPECTROPHOTOMETRIC METHODS

5.1 Thioglycolic Acid Method

5.1.1 *General*

In an alkaline medium, thioglycolic acid gives a blue to purple colour with ferric ion, a red to purple with ferrous ion. So, in practical use, it reacts in ammoniacal solution to give a reddish purple colour which is an equilibrium of ferrous and ferric compounds. The reaction is suitable for spectrophotometric measurements. The colour is stable for 12 h if protected from light, is not affected by concentration of reagent or *p*H within wide limits, and follows Beer's law. The solution has an absorption maximum at 535 nm at *p*H 10. The molecular absorption coefficient of the complex is 3 800.

Anions like fluoride, orthophosphate, tartrate, oxalate, citrate and acetate do not interfere. Metal ions like cobalt, nickel, lead, bismuth, mercury, silver and gold produce coloured complexes and thus cause interference. Some fading of colour is caused by copper, arsenic, tin, zinc and cadmium which can be overcome by adding more of the reagent.

5.1.2 *Reagents*

5.1.2.1 *Standard iron solution* — Dissolve 7.02 g of ammonium ferrous sulphate [$(NH_4)_2SO_4FeSO_4.6H_2O$] in water containing 10 ml of concentrated sulphuric acid and make up the volume with water to 1 000 ml. One millilitre of this solution contains 1.0 mg of iron (as Fe). It may be diluted suitably to contain 10, 25 or 100 µg of iron per millilitre.

5.1.2.2 *Thioglycolic acid (Mercapto — Acetic acid)* — $SH.CH_2COOH$.

5.1.2.3 *Concentrated ammonia* — Sp. Gr. 0.85 - 0.90.

5.1.2.4 *Citric acid solution* — Dissolve 20 g citric acid in water and dilute to 100 ml.

5.1.3 *Procedure*

Take several aliquots of standard iron solution (25 µg/ml), in separate 25 ml volumetric flask, add 0.5 ml of citric acid, neutralize with ammonia, add 0.1 ml of thioglycolic acid, add additional ammonia (3 ml to bring the *p*H to about 10) and make up the volume to 25 ml. After 10 min, determine the absorbance against a reagent blank at 535 nm in 1 cm cell. Draw a calibration curve by plotting concentration (in µg/ml) against absorbance.

Take a clean solution of the material prepared as prescribed in the relevant material specification, containing 0.01 to 0.2 mg of iron (as Fe). Measure an aliquot, add 0.5 ml citric acid solution, neutralize with ammonia, add 0.1 ml thioglycolic acid, followed by sufficient ammonia (3.0 ml) to render the solution alkaline. Make up the volume to 25 ml and determine the absorbance at 535 nm using a 1 cm cell. Read the concentration (in µg/ml) of the final solution from the calibration curve and calculate the total amount of iron present.

5.1.4 *Calculation*

$$\text{Iron (as Fe), ppm} = \frac{C \times 25}{W}$$

where

C = concentration of iron from the calibration curve, in µg/ml; and

W = weight, in g, of sample contained in an aliquot used for the test.

5.2 *o*-Phenanthroline Method

5.2.1 *General*

Ferrous ion reacts with *o*-phenanthroline between *p*H 3 and 9 in the ratio 1:3 to give a stable, orange-red coloured complex which has a maximum absorbance at 510 nm. The colour developed is independent of *p*H in the range 2 to 9. The molecular absorption coefficient of the complex is 11250. The molecular formula of the complex is $[(C_{12}H_8N_2)_3Fe]X_2$. Silver and bismuth give precipitate. Copper, cadmium, mercury and zinc give slightly soluble complexes and reduce the intensity of the colour. The effect of these latter ions can be overcome by using excess reagent.

5.2.2 *Reagents*

5.2.2.1 *Standard iron solutions — See* **5.1.2.1**.

5.2.2.2 *Hydroxylamine hydrochloride solution* — Dissolve 10 g in 100 ml of water and store in a dark bottle with a ground-glass stopper.

5.2.2.3 *o-Phenanthroline solution* — Dissolve 0.5 g of *o*-phenanthroline in 100 ml of water and store in a dark bottle with a ground-glass stopper.

5.2.2.4 *Sodium acetate* — Acetic acid buffer solution (*p*H 5). Prepare by mixing 300 ml of 0.2 N acetic acid and 700 ml of 0.2 M sodium acetate.

5.2.3 *Procedure*

Take several aliquots of standard iron solution (25 µg/ml) in a series of 25 ml volumetric flasks, add 2 ml of hydroxylamine hydrochloride solution, 1 ml of *o*-phenanthroline solution and adequate volume of buffer solution to bring the *p*H value to about 5. Dilute the solution in each flask to 25 ml. After 30 min, determine the absorbance against a reagent blank at 510 nm using a 1 cm cell. Draw a calibration curve between absorbance and concentration of iron (in µg/ml).

Take a clear solution of the material, prepared as prescribed in the relevant material specification, containing 0.01 to 0.2 mg of iron. To an aliquot of the sample solution, add 2 ml of hydroxylamine hydrochloride solution, 1 ml of *o*-phenanthroline solution and an adequate volume of buffer solution to bring the *p*H value to 5. Dilute to 25 ml. Determine the absorbance after 30 min against a reagent blank at 510 nm using a 1 cm cell. Read the concentration (in µg/ml) of the final solution from the calibration curve and calculate the total amount of iron present.

5.2.4 *Calculation*

$$\text{Iron (as Fe) ppm} = \frac{C \times 25}{W}$$

where

C = concentration of iron from calibration curve, in µg/ml; and

W = weight, in g, of sample contained in an aliquot used for the test.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

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Bharat Electronics Limited, Bangalore	Shri N. Ravi Bhusan
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Analytical Method Validation for Inorganic Chemicals Subcommittee, CHD 1 : 1

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This Indian Standard has been developed from Doc: No. CHD 1 (1025).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Simco Printing Press, Delhi